

**Test Material:** Dicamba

**MRID:** 49067701


**Title:** Evaporation Behavior of the Test Substance [ $^{14}\text{C}$ ]-Dicamba from Soil and Plants (Model Chamber). Test product: SAN 1411 H 167 SL 003 BS.

**EPA PC Code:** 100094

**OCSPP Guideline:** 835.1410

**For CDM Smith**

**Primary Reviewer:** Dan Hunt

**Signature:** 

**Date:** 7/8/15

**Secondary Reviewer:** Mary Samuel

**Signature:** 

**Date:** 7/8/15

**QC/QA Manager:** Joan Gaidos

**Signature:** 

**Date:** 7/8/15

## Laboratory volatility of Dicamba

**Report:** MRID 49067701. Jonas, W. 1997. Evaporation Behavior of the Test Substance [<sup>14</sup>C]-Dicamba from Soil and Plants (Model Chamber). Test product: SAN 1411 H 167 SL 003 BS. Unpublished study performed by NATEC Institut, Hamburg, Germany; sponsored and submitted by BASF Corporation, Research Triangle Park, North Carolina. Study No.: NA 96 9408/1. BASF Registration Document No. 1997/5000070. Experiment started January 20, 1997, and completed March 7, 1997 (p. 12). Final report issued June 16, 1997.

**Document No.:** MRID 49067701


**Guideline:** Conducted under: BBA Guideline Part IV, 6-1 from July 1990  
Reviewed under: OCSPP 835.1410

**Statements:** The study was conducted in compliance with the OECD Principles of GLP (1994; pp. 3, 8, 11, 13). Signed and dated Data Confidentiality, GLP, and Certificate of Authenticity statements were provided (pp. 2-3, 8-9). An unsigned Quality Assurance statement was provided (p. 10)

**Classification:** This study is invalid. The concentration of dicamba in air was not measured and the rate of volatilization could not be determined. Sampling intervals were inadequate to demonstrate the decline of the active ingredient. Test soil was not characterized and the soil was not compared to domestic (USA) soils.

**PC Code:** 100094

**Reviewer:** William P. Eckel, Ph.D.  
Senior Science Advisor

Signature:   
Date: October 13, 2015

## Executive Summary

In a laboratory study, the volatility of [<sup>14</sup>C]-dicamba from BBA standard soil 2.1 (texture and other properties not reported) and plant samples under aerobic soil conditions, at 20 ± 1°C, was investigated separately for a period of 24 hours. It could not be determined whether the study soil was representative of an intended use-site. The soil sample was treated at 0.53 mg a.i., equivalent to a field application rate of 356 g a.i./ha (0.318 lbs a.i./A), which was reported as the maximum intended field rate. A single test system was examined in the study. In a separate experiment, [<sup>14</sup>C]-dicamba was applied at a rate of 0.703 mg a.i. to two corn plants, which is equivalent to a field application rate of 305 g a.i./ha (0.272 lbs a.i./A).

Air concentrations were not determined; [<sup>14</sup>C]residues were determined from volatile traps to determine the amount of dicamba that had evaporated from the treated soil and the treated plant test system over time. At the termination of the 24-hour study period, the soil and plant material were extracted and analyzed by LSC and TLC. Post-extracted soil samples were also analyzed by LSC following combustion.

At the end of the study, a total of 1.15% of the applied radioactivity volatilized from the treated soil and 0.12% volatilized from the treated plants. Radioactivity in the soil and plant extracts was predominantly dicamba (98.1-98.4%), with other residues  $\leq 1.6\%$  (unidentified). The test system was adequate to trap the parent and its transformation products. Air concentrations and volatility estimates were not determined.

## I. Material and Methods

### A. Materials:

1. **Test Material:** The test material was prepared by combining 3.48 mg KOH, 12.8 mg surfactant, and 12.8 mg deionized water with 2.59 mg [ $^{14}\text{C}$ ]-Dicamba and 3.74 mg non-radiolabeled SAN 1367 and stirring at 50°C for 2 hr (p. 20).

**Table 1. Properties of Test Material**

Property	Value
Product Name	SAN 1411 H 167 SL 003 BS <sup>1</sup>
Formulation Type	Liquid
Typical end-use product?	Yes
Contaminants and/or impurities	Not reported
Manufacture #	Not reported
Lot #	911 115
Type of radiolabel	Not reported
Specific radioactivity	1561.4 MBq/mMole (7.065 MBq/mg)
Radiochemical purity	98-98.5% (TLC)
CAS #	1918-00-9
Chemical structure	Not reported
Storage stability	Not reported
pH	Not reported

Data obtained from pp. 12, 18-19, 25, of the study report.

<sup>1</sup> The test material contained 67 g Dicamba/L (6.3% w/w) and 100 g non-radiolabeled SAN 1367 H/L (9.4% w/w). Values in the table are provided for dicamba.

2. **Storage Conditions:** Stored in darkness (-20°C; p. 18).
3. **Soil:** German Standard Soil 2.1 was used for the soil experiment (p. 12; Appendix 1, p. 47).

**Table 2. Soil(s) Collection, Storage and Properties**

Property	Value
Geographic location	Not reported
Pesticide use history at the collection site	Not reported
Collection date	Not reported
Collection procedures	Not reported
Sampling depth	Not reported
Storage conditions	Not reported
Storage duration	Not reported

Property	Value
Soil preparation	Not reported
Soil texture (USDA <sup>1</sup> ):	Not reported
% Sand	Not reported
% Silt	Not reported
% Clay	Not reported
pH (0.01M CaCl <sub>2</sub> )	5.9
Organic carbon (%)	0.62
Organic matter (%)	Not reported
CEC (meq/100 g)	5.0
Soil Moisture Content (units):	
At 0.1 bar (pF 2.0)	Not reported
At 1/3 bar (pF 2.5)	Not reported
Bulk density (g/1000 ml)	1410
Microbial biomass (units):	
At initiation	Not reported
At termination	Not reported
Soil taxonomic classification (WRB)	Not reported

Data obtained pp. 13, 23; Appendix 1, p. 47, of the study report.

## B. Study Design:

- 1. Experimental Conditions:** The test system consisted of a glass cylinder (15 L volume, with 20.5 cm diameter and *ca.* 45 cm height) with a propeller to circulate the air (p. 21; Figure 1, p. 22). Air was continuously drawn by vacuum through the test system (26 L/min), followed by a coiled condenser and active charcoal and sodium hydroxide traps (Summarized in **Table 3**). Wind speed was measured prior to the study initiation using an anemometer; temperature and humidity were recorded using a digital thermo-hygrometer. The author stated that soil moisture was adjusted during the study period using a wick system.

**Table 3. Experimental Design**

Parameter	Soil Experiment	Corn Experiment
Duration of the test (days)	1-day	
Soil condition (Air dried/fresh)	Not reported	N/A
Soil sample weight (g/replicate)	Not reported	N/A
Soil depth (cm)	1 cm	N/A
Test concentration	0.53 mg a.i./test system	0.703 mg a.i./test system
Field Equivalent Application Rate	0.318 lb a.i./A	0.272 lb a.i./A
Number of replicates	One	One
Test apparatus	A glass cylinder containing treated soil in a glass Petri dish (0.015 m <sup>2</sup> ) was attached to a coiled condenser to collect condensed water followed by continuous flow-through volatile trapping systems containing active charcoal and sodium hydroxide. The test system is illustrated in Figure 1,	The pot containing the two treated corn plants (at the 4-leaf stage) was placed into a glass cylinder and attached to a coiled condenser to collect condensed water followed by continuous flow-through volatile trapping systems containing active

<sup>1</sup> U.S. Department of Agriculture



Parameter		Soil Experiment	Corn Experiment
		p. 22.	charcoal and sodium hydroxide. The test system is illustrated in Figure 1, p. 22.
Test material application	Test solution volume used/ treatment	0.63 mL	0.97 mL
	Application method	The application was made from a height of <i>ca.</i> 20-30 cm above the soil via a spray jet (nozzle not specified) connected to an application system constructed for application of small volumes of solutions (0.1-1 mL).	The application was made from a height of <i>ca.</i> 5-15 cm above the plant top via a spray jet (nozzle not specified) connected to an application system constructed for application of small volumes of solutions (0.1-1 mL).
Indication of test material adsorbing to walls of test apparatus?		0.81% of the applied was obtained from the chamber wash at study termination.	0.07% of the applied was obtained from the chamber wash at study termination.
Experimental conditions	Temperature (°C)	20 ± 1	20 ± 1
	Relative humidity	40 ± 1%	40 ± 1%
	Soil moisture content	60 ± 1% of maximum capacity	N/A
	Moisture maintenance method	Adjusted using a wick system	N/A
	Air flow through system	Continuous	Continuous
	Continuous darkness (Yes/No):	Not reported	Not reported
Other observations (if applicable)			Prior to the test application, the two corn plants were covered with aluminum foil and placed into the application chamber. Cultivation soil was also covered with aluminum foil and filter paper prior to the test application. The aluminum foil remained in place for the duration of the test.

Data obtained from pp. 21, 23, 26, 28, 33, 36; Figure 1, p. 22; Table 4, p. 34; Table 6, p. 37, of the study report.

2. **Sampling During Study Period:** Air samples were not collected and concentrations were not determined; [<sup>14</sup>C]residues were determined from volatile traps (Details summarized in **Table 4**). Following the 24-hour sampling interval, the test system was opened and all surfaces of the model chamber were rinsed with acetone to determine total radioactivity on the surfaces of the unit.

**Table 4. Sampling Design**

Parameter	Description
<b>Soil Experiment</b>	
Sample intervals (hours)	1, 3, 6 and 24 hours
Sampling method	Volatile traps were collected at each sampling interval.
Desired air flow of sampler (L/min)	26 L/min
Sample storage before analysis (Yes/No)?	Not reported
<b>Plant Experiment</b>	
Sample intervals (hours)	1, 3, 6 and 24 hours
Sampling method	Volatile traps were collected at each sampling interval.
Desired air flow of sampler (L/min)	26 L/min
Sample storage before analysis (Yes/No)?	Not reported

Data obtained from pp. 27, 33, 36, of the study report.

### **3. Sample Handling and Storage Stability:**

Storage conditions of samples after collection and the longest duration of storage were not reported.

### **4. Analytical Procedures:**

**Extraction methods:** Soil samples were extracted twice with 25 mL acetone:water (1:1, v:v) for 15 minutes in an ultrasonic bath and twice with acetone (all extractions were followed by centrifugation for 10 minutes at 3000 rpm; p. 29). Extracts were combined and analyzed to determine the total radioactivity. Post-extracted soil samples were air-dried and analyzed for total radioactivity following combustion.

Plants were removed, cut into small pieces with scissors, and extracted twice with 25 mL acetone:water (1:1, v:v) for 15 minutes in an ultrasonic bath and twice with acetone (all extractions were followed by centrifugation for 10 minutes at 3000 rpm; p. 29). Extracts were combined and analyzed to determine the total radioactivity. Extracted plant residues were dried, mixed with 5 mL Soluene 350, and hydrolyzed for 24 hours at 40°C prior to analysis for total radioactivity by LSC.

Active charcoal samples were homogenized and subsamples (*ca.* 50 mg) were combusted and analyzed for total radioactivity by LSC (p. 29).

Condensate water and NaOH traps were analyzed directly for total radioactivity by LSC (p. 29).

**Total Radioactivity Measurement:** Total  $^{14}\text{C}$  residues were determined by summing the percent of applied mass found in residues measured in the condensate water, charcoal and NaOH traps, chamber wash, soil/plant extracts and post-extracted soil samples.

**Identification and Quantification of Parent Compound:** Aliquots of the sorbent extracts were analyzed using TLC (silica gel 60F<sub>254</sub>, 20 x 20 cm) developed in toluene:acetone:acetic acid (65:30:5, v:v:v; p. 31). Detection was performed with a radio TLC scanner.

**Detection Limits (LOD, LOQ) for the parent compound:** The limit of detection (LOD) was determined to be 0.0005 µg for condensate water, 0.003 µg for NaOH, 0.038 µg for soil extracts, 0.022 µg for plant extracts, 0.005 µg for active charcoal combustion samples and 0.055 µg for soil combustion samples (Table 2, p. 32).

**Detection Limits (LOD, LOQ) for the Transformation Products:** Same as parent

**Instrument performance:** Not provided.

**Lab recovery, air sampling sorbent material:** Recovery of radioactivity from active charcoal fortified with [ $^{14}\text{C}$ ]dicamba was determined to be 101% in a preliminary study (pp. 24, 33).

**Lab recovery, soils:** Not performed

**Breakthrough, air samples:** N/A

## **II. Results and Discussion**

### **A. Data**

Percentage of the applied radioactivity detected in condensate water, charcoal and NaOH traps, chamber wash, soil extracts, plant extracts and post-extracted soil are shown in **Table 5**. Air concentrations were not measured in this study.

### **B. Material Balance**

The material balances for the soil and plant experiments are shown in **Table 5**. At study termination following 24 hours, total recovery from the soil experiment was 102.3% of the applied radioactivity and total recovery from the plant experiment was 100.2% (Table 4, pp. 34-35; Table 6, pp. 37-38); 98.1-98.4% of the radioactivity in the soil and plant extracts was identified as parent (Figures 5-6, pp. 43-44). The chamber wash accounted for  $\leq 0.81\%$  of the applied radioactivity.

### **C. Microbial Activity**

Microbial biomass in the test soil was not monitored during the study. Soil moisture was not reported; however, it was stated that the moisture level was adjusted during the study period (p. 23). Temperature was recorded but values were not reported (p. 21).

### **D. Transformation Products**

Air concentrations were not determined. An unidentified transformation product accounted for 1.59% of the radioactivity contained in the soil extract and two unidentified transformation products accounted for  $\leq 1.15\%$  of the radioactivity in the plant extract (Figures 5-6, pp. 43-44).

### **E. Volatilization**

Volatilization flux rates were not determined.

<b>Table 5a. Volatilization of [<sup>14</sup>C]Dicamba, expressed as a percentage of the applied, from soil.</b>					
<b>Sampling Interval</b>	<b>0-1 hr</b>	<b>1-3 hr</b>	<b>3-6 hr</b>	<b>6-24 hr</b>	<b>Sum</b>
Condensate water	0.25	0.01	0.01	0.04	0.32
Active charcoal	<0.01	<0.01	<0.01	<0.01	<0.01
Cotton stoppers	<0.01	<0.01	<0.01	<0.01	<0.01
Sodium hydroxide	<0.01	<0.01	<0.01	0.01	0.01
Chamber wash					0.81
<b>Sum (evaporated)</b>					<b>1.15</b>
Soil (extract)					92.67
Soil (combusted)					8.50
<b>Sum (soil)</b>					<b>101.1</b>
<b>Mass balance</b>					<b>102.3</b>

Data obtained from Table 4, pp. 34-35, of the study report. Radioactivity in the soil extract was identified as dicamba (98.4%; Figure 5, p. 43).

<b>Table 5b. Volatilization of [<sup>14</sup>C]Dicamba, expressed as a percentage of the applied, from plants.</b>					
<b>Sampling Interval</b>	<b>0-1 hr</b>	<b>1-3 hr</b>	<b>3-6 hr</b>	<b>6-24 hr</b>	<b>Sum</b>
Condensate water	0.03	<0.01	0.01	0.01	0.05
Active charcoal	<0.01	<0.01	<0.01	<0.01	<0.01
Cotton stoppers	<0.01	<0.01	<0.01	<0.01	<0.01
Sodium hydroxide	<0.01	<0.01	<0.01	<0.01	<0.01
Chamber wash					0.07
<b>Sum (evaporated)</b>					<b>0.12</b>
Plant (extract)					99.19
Plant (hydrolysis with Soluene)					0.91
<b>Sum (soil)</b>					<b>100.1</b>
<b>Mass balance</b>					<b>100.2</b>

Data obtained from Table 6, pp. 37-38, of the study report. Radioactivity in the soil extract was identified as dicamba (98.1%; Figure 6, p. 44).



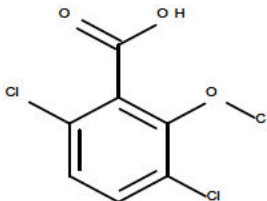
### III. Study Deficiencies and Reviewer's Comments

1. The concentration of dicamba in air was not measured and the rate of volatilization could not be determined.
2. Sampling intervals were inadequate to demonstrate the decline of the active ingredient. The study was only conducted for 24 hours and  $\leq 1.15\%$  of the applied radioactivity was shown to have evaporated during the study period (Table 4, pp. 34-35; Table 6, pp. 37-38). .
3. Test soil was not characterized according to the USDA soil classification system and the soil was not compared to domestic (USA) soils. Soil particle size class data were provided in Appendix 1 (p. 47) of the study report; however, the soil texture could not be determined because the particle size analysis was not conducted according to the USDA classification system. A sandy soil is recommended for use in this study type. It was not specified whether the soil was sieved prior to use.
4. The length of storage of test samples and sample extracts was not reported and a storage stability study was not conducted to determine the stability of dicamba in the traps and extracts.
5. The experiments were not replicated.
6. It was not specifically stated that the experiments were conducted in darkness.
7. The vapor pressure of dicamba was not reported.

### IV. References

1. U.S. Environmental Protection Agency. 2008. Fate, Transport and Transformation Test Guidelines, OCSPP 835.1410, Laboratory Volatility. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-08-011.
2. U.S. Environmental Protection Agency (USEPA). 1989. FIFRA Accelerated Reregistration, Phase 3 Technical Guidance. Office of the Prevention, Pesticides, and Toxic Substances, Washington, DC. EPA 540/09-90-078.

**DER ATTACHMENT 1. Dicamba and Its Environmental Transformation Products. <sup>A</sup>**

Code Name/ Synonym	Chemical Name	Chemical Structure	Study Type	MRID	Maximum %AR (day)	Final %AR (study length)
<b>PARENT</b>						
<b>Dicamba</b> <b>(SAN 1367 H</b> <b>A1; BAS183</b> <b>22 H)</b>	<b>IUPAC:</b> 3,6-Dichloro-o-anisic acid  <b>CAS:</b> 3,6-Dichloro-2-methoxybenzoic acid  <b>CAS No.:</b> 1918-00-9  <b>Formula:</b> $C_8H_6Cl_2O_3$ <b>MW:</b> 221.04 g/mol <b>SMILES:</b> <chem>COc1c(Cl)ccc(Cl)c1C(=O)O</chem>		835.1410 Laboratory Volatility	490677 01	PRT	PRT
<b>MAJOR (&gt;10%) TRANSFORMATION PRODUCTS</b>						
<b>No major transformation products were identified.</b>						
<b>MINOR (&lt;10%) TRANSFORMATION PRODUCTS</b>						
<b>No minor transformation products were identified.</b>						
<b>REFERENCE COMPOUNDS NOT IDENTIFIED</b>						
<b>All compounds used as reference compounds were identified.</b>						

<sup>A</sup> AR means “applied radioactivity”. MW means “molecular weight”. PRT means “parent”.